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Biler et al.

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(54) **SOLID ELECTROLYTIC CAPACITOR**

USPC 361/503-504, 509, 516-519, 523, 525,
361/528-529, 535-536

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See application file for complete search history.

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(51) **Int. Cl.**

(57) **ABSTRACT**

H01G 9/028	(2006.01)
H01G 9/052	(2006.01)
H01G 9/00	(2006.01)
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H01G 9/15	(2006.01)

A capacitor for use in relatively high voltage environments is provided. The solid electrolyte is formed from a plurality of pre-polymerized particles in the form of a dispersion. In addition, the anode is formed such that it contains at least one longitudinally extending channel is recessed therein. The channel may have a relatively high aspect ratio (length divided by width), such as about 2 or more, in some embodiments about 5 or more, in some embodiments from about 10 to about 200, in some embodiments from about 15 to about 150, in some embodiments from about 20 to about 100, and in some embodiments, from about 30 to about 60.

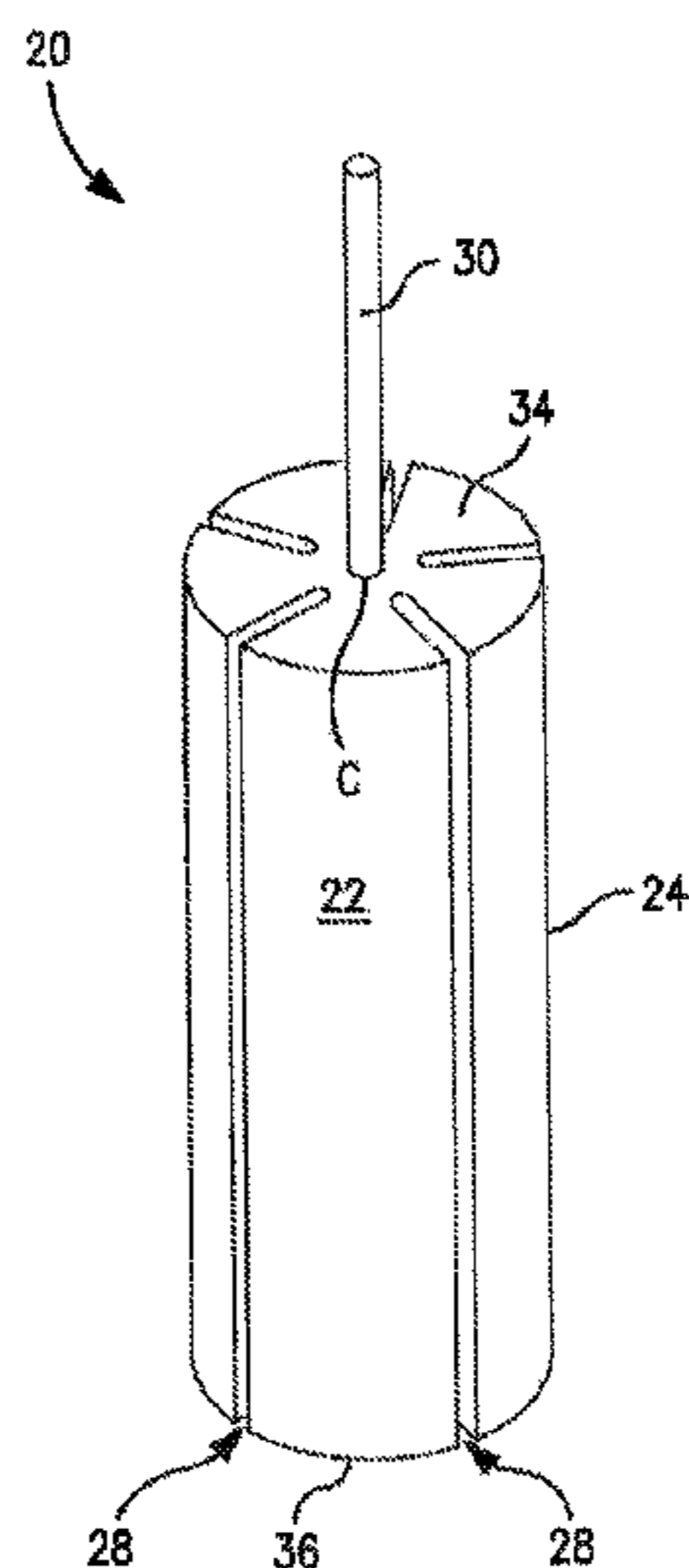
(52) **U.S. Cl.**

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(2013.01)

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H01G 9/0029

20 Claims, 3 Drawing Sheets



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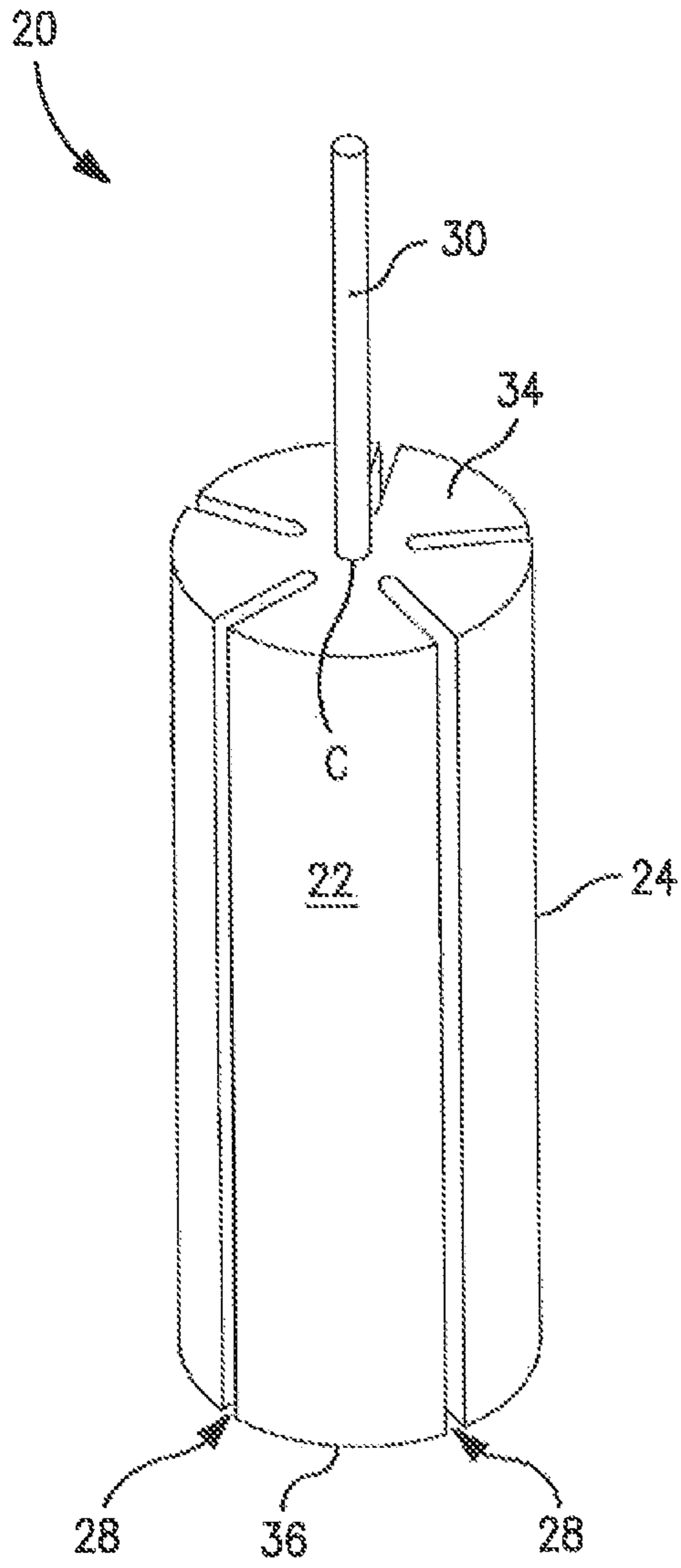


FIG. 1

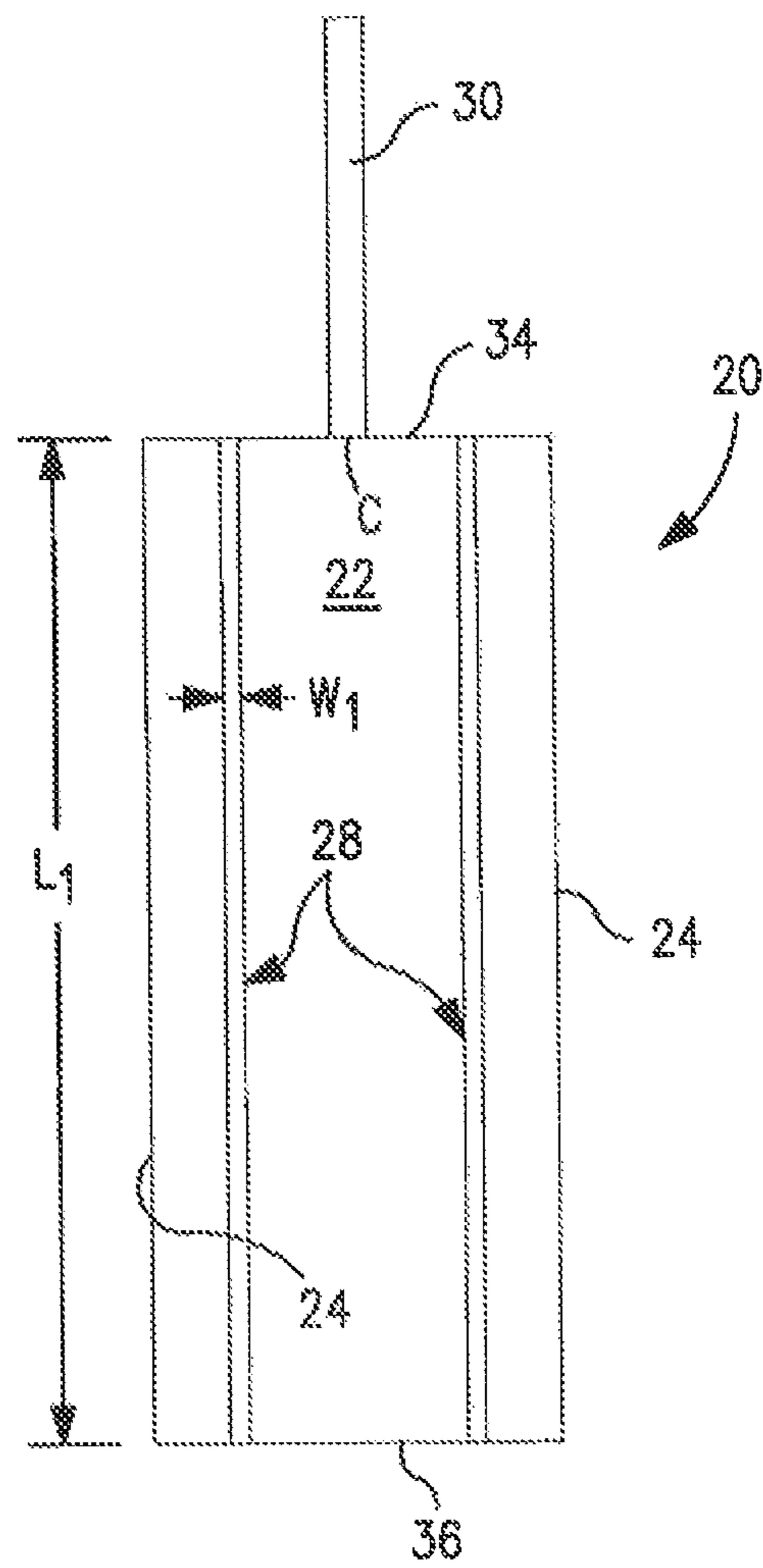
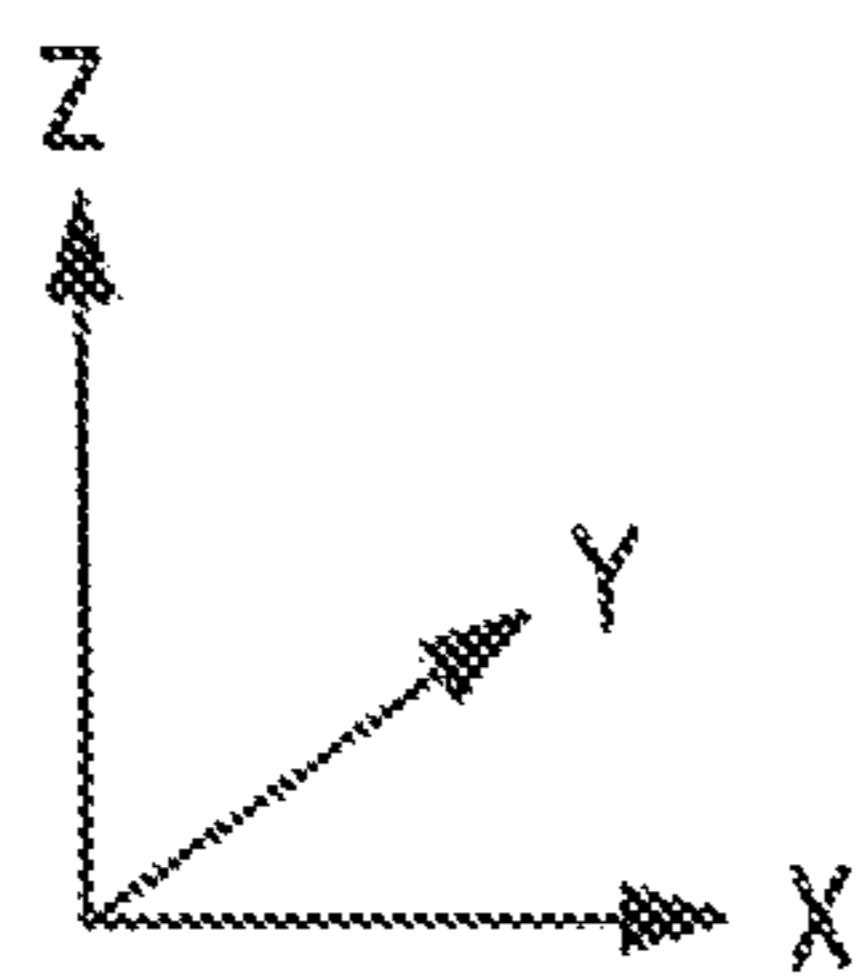


FIG. 2

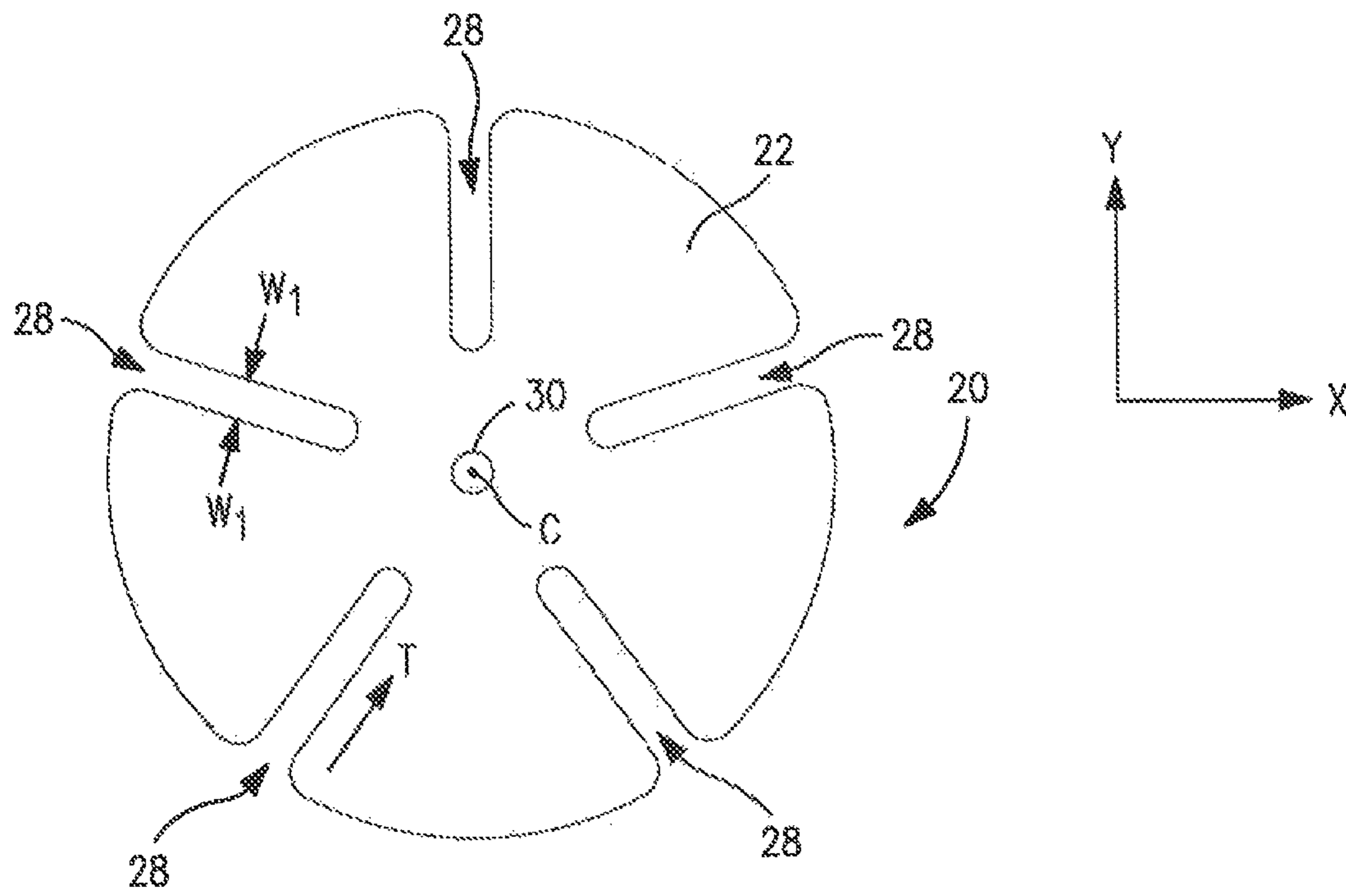


FIG. 3

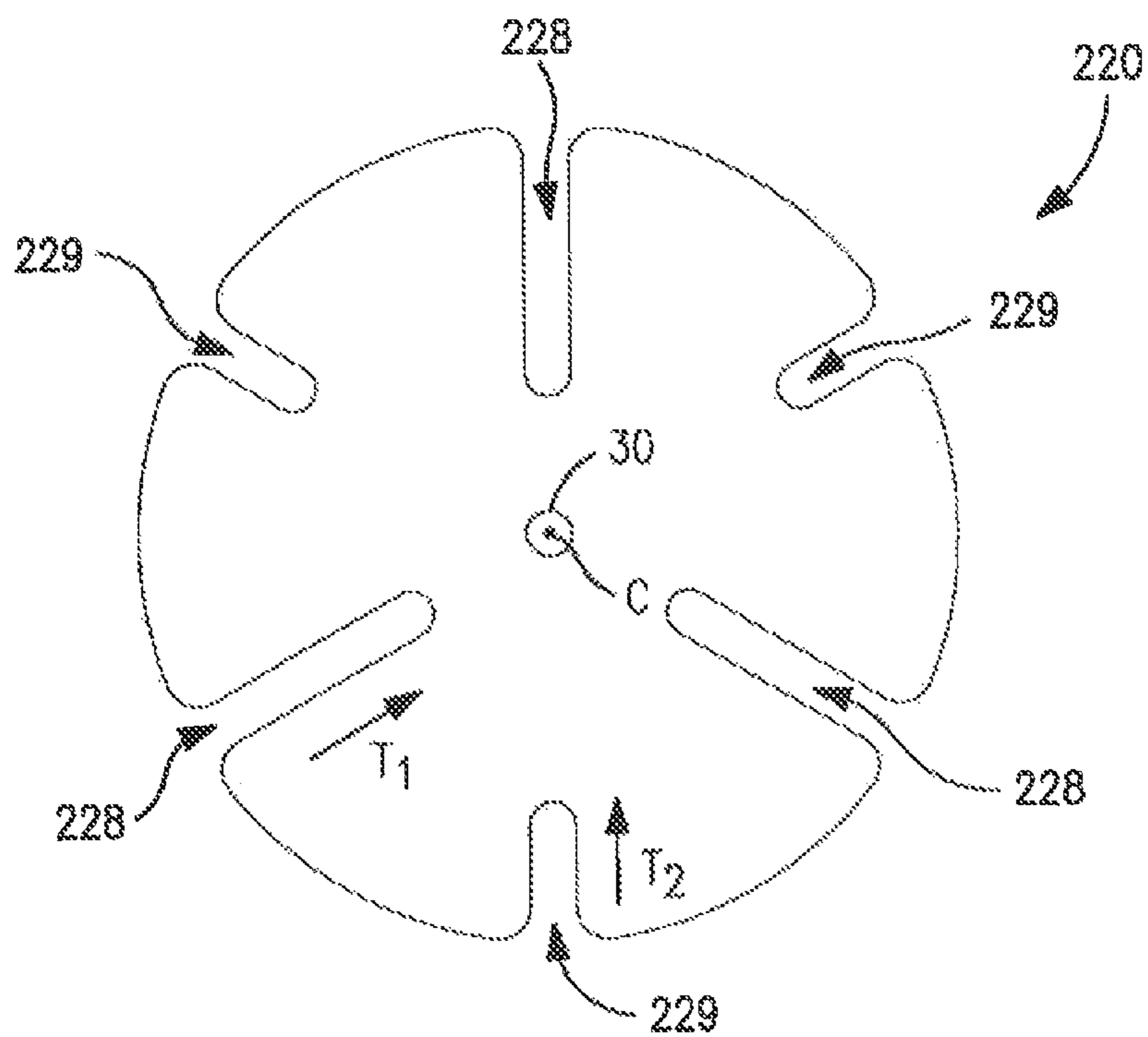


FIG. 4

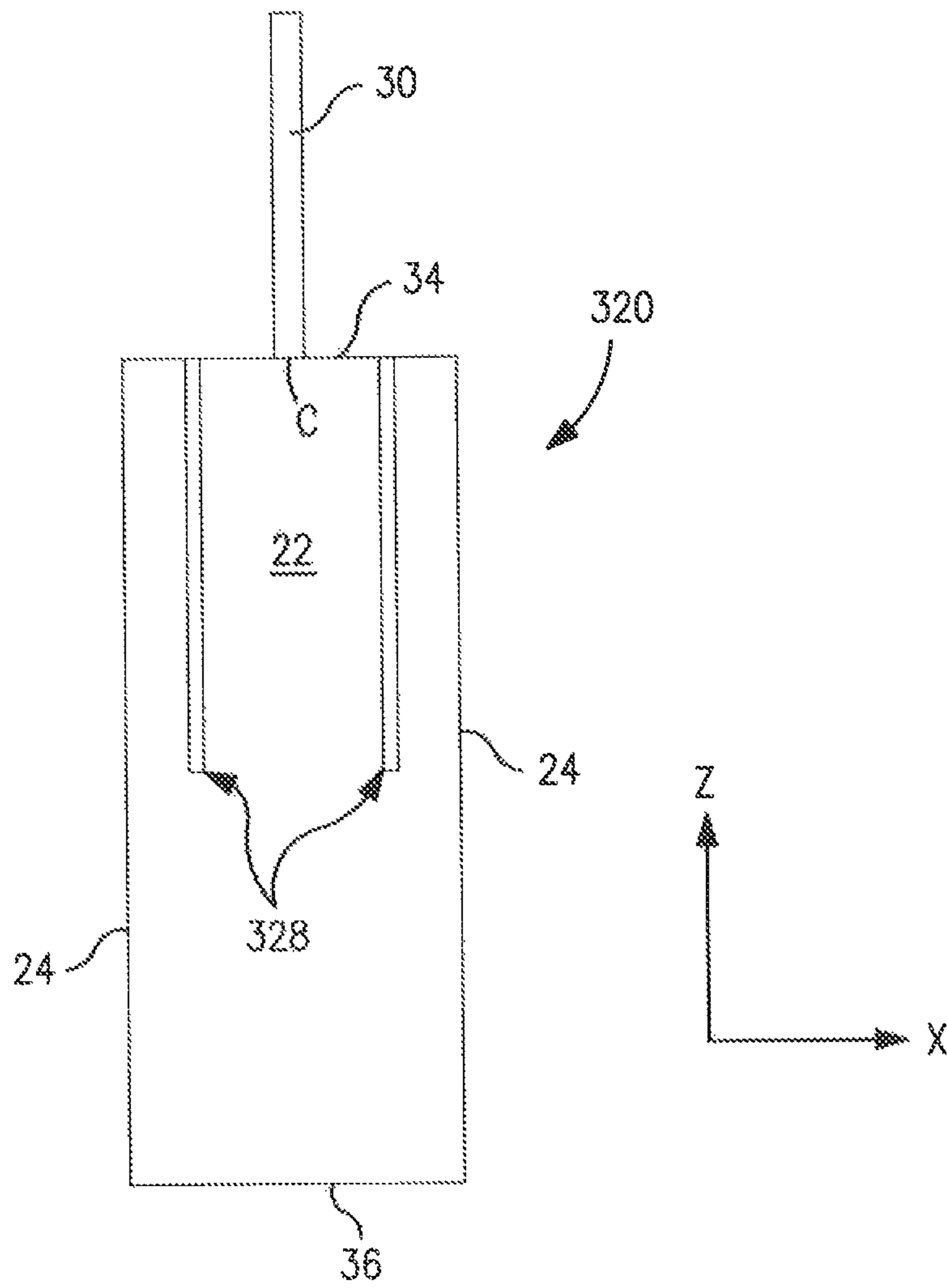


FIG. 5

1

SOLID ELECTROLYTIC CAPACITOR

RELATED APPLICATIONS

The present application claims priority to U.S. Provisional Application Ser. No. 61/787,015 (filed on Mar. 15, 2013) and which is incorporated herein in its entirety by reference thereto.

BACKGROUND OF THE INVENTION

Solid electrolytic capacitors (e.g., tantalum capacitors) are typically made by pressing a metal powder (e.g., tantalum) around a metal lead wire, sintering the pressed part, anodizing the sintered anode, and thereafter applying a solid electrolyte. Intrinsically conductive polymers are often employed as the solid electrolyte due to their advantageous low equivalent series resistance (“ESR”) and “non-burning/non-ignition” failure mode. Such electrolytes can be formed through in situ chemical polymerization of the monomer in the presence of a catalyst and dopant. One of the problems with conventional capacitors that employ in situ polymerized polymers is that they tend to fail at high voltages, such as experienced during a fast switch on or operational current spike. In an attempt to overcome some of these issues, premade conductive polymer slurries have also been employed in certain applications as an alternative solid electrolyte material. While some benefits have been achieved with these capacitors in high voltage environments, problems nevertheless remain. For instance, one problem with polymer slurry-based capacitors is that they can achieve only a relatively low percentage of their wet capacitance, which means that they have a relatively large capacitance loss and/or fluctuation in the presence of atmosphere humidity.

As such, a need currently exists for a solid electrolytic capacitor having improved performance in high voltage environments.

SUMMARY OF THE INVENTION

In accordance with one embodiment of the present invention, a solid electrolytic capacitor is disclosed that comprises an anode that comprises a sintered porous anode body, a dielectric overlying the anode body, and a solid electrolyte overlying the dielectric. The anode body extends in a longitudinal direction and contains a sidewall positioned between a proximal end and an opposing distal end, wherein a longitudinally extending channel is recessed into the sidewall. The solid electrolyte contains a plurality of pre-polymerized conductive polymer particles.

Other features and aspects of the present invention are set forth in greater detail below.

BRIEF DESCRIPTION OF THE DRAWINGS

A full and enabling disclosure of the present invention, including the best mode thereof to one skilled in the art, is set forth more particularly in the remainder of the specification, including reference to the accompanying figures, in which;

FIG. 1 is a perspective view of one embodiment of an anode that may be employed in the solid electrolytic capacitor of the present invention;

FIG. 2 is a side view of the anode of FIG. 1;

FIG. 3 is a top view of the anode of FIG. 1;

FIG. 4 is a top view of another embodiment of an anode that may be employed in the solid electrolytic capacitor of the present invention; and

2

FIG. 5 is a side view of yet another embodiment of an anode that may be employed in the solid electrolytic capacitor of the present invention.

Repeat use of reference characters in the present specification and drawings is intended to represent the same or analogous features or elements of the present invention.

DETAILED DESCRIPTION OF REPRESENTATIVE EMBODIMENTS

It is to be understood by one of ordinary skill in the art that the present discussion is a description of exemplary embodiments only, and is not intended as limiting the broader aspects of the present invention.

Generally speaking, the present invention is directed to a capacitor for use in relatively high voltage environments. The present inventor has discovered that the ability to achieve such voltages can be achieved through a unique and controlled combination of features relating to the formation of the anode, dielectric, and solid electrolyte. For example, the solid electrolyte is formed from a plurality of pre-polymerized particles in the form of a dispersion. In this manner, the electrolyte may remain generally free of high energy radicals (e.g. Fe^{2+} or Fe^{3+} ions) that can lead to dielectric degradation, particularly at ultrahigh voltages. In addition, the anode body is formed such that it contains at least one longitudinally extending channel is recessed therein. The channel may have a relatively high aspect ratio (length divided by width), such as about 2 or more, in some embodiments about 5 or more, in some embodiments from about 10 to about 200, in some embodiments from about 15 to about 150, in some embodiments from about 20 to about 100, and in some embodiments, from about 30 to about 60. Such channels can significantly increase the outer surface area of the anode body, which may enhance the degree to which it can dissipate heat. This may, in turn, help protect the anode body at those external locations where oxide flaws are more vulnerable to current surges experienced at high voltages.

The increased available surface area of the anode body can also increase the likelihood that the solid electrolyte will pass into its pores when applied. Alternatively, larger capacitor sizes can be achieved with a higher dielectric quality, and thus the capacitor can exhibit an increased breakdown voltage (voltage at which the capacitor fails) for use in high voltage applications. The capacitor may, for example, exhibit a relatively high “breakdown voltage” (voltage at which the capacitor fails), such as about 60 volts or more, in some embodiments about 70 volts or more, and in some embodiments about 80 volts or more. Likewise, the capacitor may also be able to withstand relatively high surge currents, which is also common in high voltage applications. The peak surge current may be, for example, about 100 Amps or more, in some embodiments about 200 Amps or more, and in some embodiments, and in some embodiments, from about 300 Amps to about 800 Amps. The capacitor can also exhibit a relatively high percentage of its wet capacitance, which enables it to have only a small capacitance loss and/or fluctuation in the presence of atmosphere humidity. This performance characteristic is quantified by the “wet-to-dry capacitance percentage”, which is determined by the equation:

$$\text{Wet-to-Dry Capacitance} = (\text{Dry Capacitance} / \text{Wet Capacitance}) \times 100$$

The capacitor of the present invention, for instance, may exhibit a wet-to-dry capacitance percentage of about 50% or

more, in some embodiments about 60% or more, in some embodiments about 70% or more, and in some embodiments, from about 80% to 100%.

The present inventor has also unexpectedly discovered that the channels of the anode can provide a multitude of other benefits to the electrical performance of the capacitor. For instance, binders and/or lubricants are often incorporated into the anode body to facilitate pressing. Although such components may be removed through various heating and/or washing steps, some residual amount of carbon can nevertheless remain in the pressed body. This residual carbon can, however, lead to crystallization of the otherwise amorphous dielectric layer formed during anodic oxidation, which can damage the anode. Although this damage may not have an appreciable effect on all types of capacitors, the present inventor has discovered that it is particularly problematic for anodes used in solid electrolytic capacitors, which are sometimes formed from powders having a high specific charge. Nevertheless, the present inventor has discovered that the channels can help reduce carbon content and thus improve electrical performance. More specifically, it is believed that the channels can create a capillary pressure that helps overcome the adhesion forces of a washing liquid and the surface of the anode body, which further increases the degree to which the washing liquid, and in turn carbon, can be removed from the anode. The resulting anode may, for example, contain less than about 100 parts per million (“ppm”) of carbon, in some embodiments less than about 50 ppm carbon, and in some embodiments, from 0 to 10 ppm carbon. The capillary pressure formed by the channels can also have other benefits. For example, the channels can allow the anodizing electrolyte to be more quickly removed upon completion of an anodization step, which enhances the rate of cooling and minimizes the formation of defects.

The anode body may also have a relatively low oxygen content. For example, the anode may have no more than about 3500 ppm oxygen, in some embodiments no more than about 3000 ppm oxygen, and in some embodiments, from about 500 to about 2500 ppm oxygen. Oxygen content may be measured by LECO Oxygen Analyzer and includes oxygen in natural oxide on the tantalum surface and bulk oxygen in the tantalum particles. Bulk oxygen content is controlled by period of crystalline lattice of tantalum, which is increasing linearly with increasing oxygen content in tantalum until the solubility limit is achieved. This method was described in “Critical Oxygen Content In Porous Anodes Of Solid Tantalum Capacitors”, Pozdeev-Freeman et al., *Journal of Materials Science: Materials In Electronics* 9, (1998) 309-311 wherein X-ray diffraction analysis (XRDA) was employed to measure period of crystalline lattice of tantalum. Oxygen in sintered tantalum anodes may be limited to thin natural surface oxide, while the bulk of tantalum is practically free of oxygen.

Various embodiments of the present invention will now be described in more detail.

I. Anode

The anode body of the anode is formed from a valve metal composition. The specific charge of the composition may vary, such as from about 2,000 $\mu\text{F}\cdot\text{V}/\text{g}$ to about 150,000 $\mu\text{F}\cdot\text{V}/\text{g}$, in some embodiments from about 3,000 $\mu\text{F}\cdot\text{V}/\text{g}$ to about 70,000 $\mu\text{F}\cdot\text{V}/\text{g}$ or more, and in some embodiments, from about 4,000 to about 50,000 $\mu\text{F}\cdot\text{V}/\text{g}$. As is known in the art, the specific charge may be determined by multiplying capacitance by the anodizing voltage employed, and then dividing this product by the weight of the anodized electrode body.

The valve metal composition generally contains a valve metal (i.e., metal that is capable of oxidation) or valve metal-

based compound, such as tantalum, niobium, aluminum, hafnium, titanium, alloys thereof, oxides thereof, nitrides thereof, and so forth. For example, the valve metal composition may contain an electrically conductive oxide of niobium, such as niobium oxide having an atomic ratio of niobium to oxygen of 1:1.0 \pm 1.0, in some embodiments 1:1.0 \pm 0.3, in some embodiments 1:1.0 \pm 0.1, and in some embodiments, 1:1.0 \pm 0.05. The niobium oxide may be NbO_{0.7}, NbO_{1.0}, NbO_{1.1}, and NbO₂. Examples of such valve metal oxides are described in U.S. Pat. No. 6,322,912 to Fife; U.S. Pat. No. 6,391,275 to Fife et al.; U.S. Pat. No. 6,416,730 to Fife et al.; U.S. Pat. No. 6,527,937 to Fife; U.S. Pat. No. 6,576,099 to Kimmel, et al.; U.S. Pat. No. 6,592,740 to Fife, et al.; and U.S. Pat. No. 6,639,787 to Kimmel, et al.; and U.S. Pat. No. 7,220,397 to Kimmel, et al., as well as U.S. Patent Application Publication Nos. 2005/0019581 to Schnitter; 2005/0103638 to Schnitter, et al.; 2005/0013765 to Thomas, et al.

To form the anode body, a powder of the valve metal composition is generally employed. The powder may contain particles any of a variety of shapes, such as nodular, angular, flake, etc., as well as mixtures thereof. In particular embodiments, the particles can have a flake-like morphology in that they possess a relatively flat or platelet shape. Such particles can provide a short transmission line between the outer surface and interior of the anode and also provide a highly continuous and dense wire-to-anode connection with high conductivity. Among other things, this may help increase the breakdown voltage (voltage at which the capacitor fails) and help lower equivalent series resistance (“ESR”). The particles may also increase the specific charge of the anode when anodized at higher voltages, thereby increasing energy density.

When employed, the flake particles are generally flat. The degree of flatness is generally defined by the “aspect ratio”, i.e., the average diameter or width of the particles divided by the average thickness (“ENT”). For example, the aspect ratio of the particles may be from about 2 to about 100, in some embodiments from about 3 to about 50, in some embodiments, from about 4 to about 30. The particles may also have a specific surface area of from about 0.5 to about 10.0 m²/g, in some embodiments from about 0.7 to about 5.0 m²/g, and in some embodiments, from about 1.0 to about 4.0 m²/g. The term “specific surface area” generally refers to surface area as determined by the physical gas adsorption (B.E.T.) method of Bruanauer, Emmet, and Teller, *Journal of American Chemical Society*, Vol. 60, 1938, p. 309, with nitrogen as the adsorption gas. The test may be conducted with a MONOSORB® Specific Surface Area Analyzer available from QUANTACHROME Corporation, Syosset, N.Y., which measures the quantity of adsorbate nitrogen gas adsorbed on a solid surface by sensing the change in thermal conductivity of a flowing mixture of adsorbate and inert carrier gas (e.g., helium).

The bulk density (also known as Scott density) is also typically from about 0.1 to about 2 grams per cubic centimeter (g/cm³), in some embodiments from about 0.2 g/cm³ to about 1.5 g/cm³, and in some embodiments, from about 0.4 g/cm³ to about 1 g/cm³. “Bulk density” may be determined using a flow meter funnel and density cup. More specifically, the powder sample may be poured through the funnel into the cup until the sample completely fills and overflows the periphery of the cup, and thereafter sample may be leveled-off by a spatula, without jarring, so that it is flush with the top of the cup. The leveled sample is transferred to a balance and weighed to the nearest 0.1 gram to determine the density value. Such an apparatus is commercially available from Alcan Aluminum Corp. of Elizabeth, N.J. The particles may also have an average size (e.g., width) of from about 0.1 to

5

about 100 micrometers, in some embodiments from about 0.5 to about 70 micrometers, and in some embodiments, from about 1 to about 50 micrometers.

Certain additional components may also be included in the powder. For example, the powder may be optionally mixed with a binder and/or lubricant to ensure that the particles adequately adhere to each other when pressed to form the anode body. Suitable binders may include, for instance, poly(vinyl butyral); poly(vinyl acetate); poly(vinyl alcohol); poly(vinyl pyrrolidone); cellulosic polymers, such as carboxymethylcellulose, methyl cellulose, ethyl cellulose, hydroxyethyl cellulose, and methylhydroxyethyl cellulose; atactic polypropylene, polyethylene; polyethylene glycol (e.g., Carbowax from Dow Chemical Co.); polystyrene, poly(butadiene/styrene); polyamides, polyimides, and polyacrylamides, high molecular weight polyethers; copolymers of ethylene oxide and propylene oxide; fluoropolymers, such as polytetrafluoroethylene, polyvinylidene fluoride, and fluoroolefin copolymers; acrylic polymers, such as sodium polyacrylate, poly(lower alkyl acrylates), poly(lower alkyl methacrylates) and copolymers of lower alkyl acrylates and methacrylates; and fatty acids and waxes, such as stearic and other soapy fatty acids, vegetable wax, microwaxes (purified paraffins), etc. The binder may be dissolved and dispersed in a solvent. Exemplary solvents may include water, alcohols, and so forth. When utilized, the percentage of binders and/or lubricants may vary from about 0.1% to about 8% by weight of the total mass. It should be understood, however, that binders and/or lubricants are not necessarily required in the present invention.

The resulting powder may then be compacted to form a pellet using any conventional powder press device. For example, a press mold may be employed that is a single station compaction press containing a die and one or multiple punches. Alternatively, anvil-type compaction press molds may be used that use only a die and single lower punch. Single station compaction press molds are available in several basic types, such as cam, toggle/knuckle and eccentric/crank presses with varying capabilities, such as single action, double action, floating die, movable platen, opposed ram, screw, impact, hot pressing, coining or sizing. The powder may be compacted around an anode lead (e.g., tantalum wire). It should be further appreciated that the anode lead may alternatively be attached (e.g., welded) to the anode body subsequent to pressing and/or sintering of the anode body. Although not required, the channels of the present invention may be formed during pressing as would be known to those skilled in the art. For example, the press mold may contain one or more longitudinal indentations that correspond to the desired shape of the channels. In this manner, the powder is compressed around the indentations so that when removed from the mold, the resulting anode body contains longitudinal channels at those areas where the longitudinal indentations were located in the mold.

After compaction, the resulting anode body may then be diced into any desired shape, such as square, rectangle, circle, oval, triangle, hexagon, octagon, heptagon, pentagon, etc. The anode body may then be subjected to a heating step in which most, if not all, of any binder/lubricant are removed. For example, the anode body is typically heated by an oven that operates at a temperature of from about 150° C. to about 500° C. Alternatively, the binder/lubricant may also be removed by contacting the pellet with an aqueous solution, such as described in U.S. Pat. No. 6,197,252 to Bishop, et al. As indicated above, the channels of the present invention can create a capillary pressure that can help increase the degree to which the liquid solution, and in turn carbon, can be removed

6

from the anode body. Thereafter, the anode body is sintered to form a porous, integral mass. The temperature, atmosphere, and time of the sintering may depend on a variety of factors, such as the type of anode, the size of the anode, etc. Typically, sintering occurs at a temperature of from about 800° C. to about 1900° C., in some embodiments from about 1000° C. to about 1500° C., and in some embodiments, from about 1100° C. to about 1400° C., for a time of from about 5 minutes to about 100 minutes, and in some embodiments, from about 30 minutes to about 60 minutes. If desired, sintering may occur in an atmosphere that limits the transfer of oxygen atoms to the anode. For example, sintering may occur in a reducing atmosphere, such as in a vacuum, inert gas, hydrogen, etc. The reducing atmosphere may be at a pressure of from about 10 Torr to about 2000 Torr, in some embodiments from about 100 Torr to about 1000 Torr, and in some embodiments, from about 100 Torr to about 930 Torr. Mixtures of hydrogen and other gases (e.g., argon or nitrogen) may also be employed.

Upon sintering, an anode body is thus formed that contains longitudinal channels in accordance with the present invention. Referring to FIGS. 1-3, for example, one embodiment of an anode **20** is shown that contains a porous, sintered body **22** having at least one sidewall **24** positioned between a proximal end **34** and an opposing distal end **36**. The cross-sectional shape of the proximal end **34** and/or the distal end **36** may generally vary based on the desired shape of the anode body **22**. In this particular embodiment, for example, both ends **34** and **36** have a circular cross-sectional shape such that the anode body **22** is generally cylindrical. Other suitable shapes may include, for instance, square, rectangular, triangular, hexagonal, octagonal, heptagonal, pentagonal, trapezoidal, elliptical, star, sinusoidal, etc.

The anode body **22** also has a length in the longitudinal direction “z” defined between the ends **34** and **36**, and a width in the “x” direction and depth in the “y” direction. In the illustrated embodiment, the width and depth are both defined between the sidewalls **24**. Although by no means a requirement, the length of the anode body **22** is typically greater than its width and/or depth. For example, in certain embodiments, the ratio of the length to both the width and depth may be from about 1 to about 30, in some embodiments from about 1.1 to about 10, and in some embodiments, from about 1.5 to about 5. The length of the anode **20** may, for example, range from about 0.5 to about 100 millimeters, in some embodiments from about 1 to about 60 millimeters, and in some embodiments, from about 5 to about 30 millimeters. The width of the anode body **22** may range from about 0.5 to about 50 millimeters, in some embodiments from about 1 to about 40 millimeters, and in some embodiments, from about 4 to about 30 millimeters. Likewise, the depth of the anode body **22** may range from about 0.5 to about 50 millimeters, in some embodiments from about 1 to about 40 millimeters, and in some embodiments, from about 4 to about 30 millimeters. Of course, when the anode body is cylindrical in nature, its width and depth will be the same.

Regardless of its particular size or shape, the anode body **22** contains channels **28** that are recessed into the sidewall **24**. The channels **28** are “longitudinally extending” in the sense that they possess a length in the longitudinal direction “z” of the anode body **22**. However, while the channels **28** of FIGS. 1-3 are substantially parallel with the longitudinal direction, this is by no means a requirement. For example, other suitable embodiments may include one or more longitudinally extending channels that are in the form of a spiral, helix, etc., which are not parallel with the longitudinal of the anode body.

The number of such longitudinally extending channels may vary, but is typically from 1 to 20, in some embodiments from 2 to 15, and in some embodiments, from 4 to 10. When multiple channels are employed, it is generally desired that they are distributed symmetrically and equidistant about a center longitudinal axis of the anode, although this is by no means a requirement. In FIGS. 1-3, for example, the depicted anode body 22 contains five (5) separate channels 28. FIG. 4, on the other hand, shows an alternative embodiment in which six (6) separate channels 228 are employed. In each of the particular embodiments, however, the channels are distributed in a generally symmetric manner about the longitudinal center "C" of the anode body.

As indicated above, at least a portion of the channels 28 have a relatively high aspect ratio (length divided by width). The length "L₁" (FIG. 2) of the channels 28 may, for example, range from about 0.5 to about 100 millimeters, in some embodiments from about 1 to about 60 millimeters, and in some embodiments, from about 5 to about 30 millimeters. The width "W₁" of the channels 28 (FIGS. 2 and 3) may likewise range from about 0.01 to about 20 millimeters, in some embodiments from about 0.02 to about 15 millimeters, in some embodiments from about 0.05 to about 4 millimeters, and in some embodiments, from about 0.1 to about 2 millimeters.

The channels 28 shown in FIGS. 1-3 extend in the longitudinal direction "L" along the entire length of the anode body 22 and intersect both the proximal end 34 and the distal end 36. It should be understood, however, that one or more channels may also extend along only a portion of the anode body length so that they intersect only one end of the anode body, or so that they do not intersect either end. Referring to FIG. 5, for instance, one embodiment of such an anode 320 is shown that contains a plurality of longitudinally extending channels 328 that are recessed into a sidewall 24, but do not intersect either a proximal end 34 or distal end 36 of the anode 320. Generally speaking, however, the ratio of the length of the anode body to the length of the channels in the longitudinal direction is from about 0.2 to 1, in some embodiments from about 0.5 to 1, and in some embodiments, from about 0.8 to 1. The ratio of the width of the anode body (e.g., diameter) to the width of the channels may also be within a range of from about 1 to about 100, in some embodiments from about 2 to about 40, and in some embodiments, from about 5 to about 30. In the embodiment shown in FIGS. 1-3, the cross-sectional width of the channels 28 is substantially constant and of a generally rectangular shape. Nevertheless, it should be understood that any other shape may also be employed, and that the width and/or shape of the channels may vary along the length of the channels if desired.

The extent to which the channels are recessed into the anode body may be selectively controlled in the present invention to achieve a balance between increased surface and integrity of the anode structure. That is, if the depth of the channels is too great, it may be difficult to press the anode into a physically strong structure. Likewise, if the depth is too small, the desired benefits may not be achieved. Thus, in most embodiments, the channels are recessed so that they extend in a direction that is from about 2% to about 60%, in some embodiments from about 5% to about 50%, and in some embodiments, from about 10% to about 45% of the thickness of the anode body in the same direction. Referring again to FIG. 3, for example, one of the channels 28 is shown as extending in a direction "T." In this embodiment, the length of the channel 28 in the direction "T" divided by the thickness of the porous body 22 in the direction "T", multiplied by 100, is within the percentages referenced above.

Of course, the depth of each of the channels need not be the same. Referring to FIG. 4, for example, one embodiment of an anode 220 is shown that contains first channels 228 and second channels 229. In this particular embodiment, the first channels 228 extend into the anode body to a greater degree than the second channels 229. One of the first channels 228 may, for example, extend in a direction "T₁" that is from about 15% to about 60%, in some embodiments from about 20% to about 50%, and in some embodiments, from about 25% to about 45% of the thickness of the anode body in the same direction. Likewise, one of the second channels 229 may extend in a direction "T₂" that is from about 2% to about 40%, in some embodiments from about 5% to about 35%, and in some embodiments, from about 10% to about 25% of the anode body in the same direction. Such a configuration can effectively combine the benefits of the deeper channels (e.g., greater surface area) with those of the shallower channels (e.g., greater physical integrity). In such embodiments, the number of deeper channels may be from 1 to 10, in some embodiments from 2 to 6, and in some embodiments, from 2 to 4, and the number of shallower channels may likewise be from 1 to 10, in some embodiments from 2 to 6, and in some embodiments, from 2 to 4.

Typically, the anode of the present invention also contains an anode lead wire that helps connect the anode to the terminations of the resulting capacitor. The lead wire may be formed from any electrically conductive material, such as tantalum, niobium, nickel, aluminum, hafnium, titanium, etc., as well as oxides and/or nitrides of thereof. Although not necessarily required, it is often desired that the lead wire extend in the same longitudinal direction as the channels. In the embodiment of FIGS. 1-3, for example, an anode lead wire 30 extends in the longitudinal "z" direction from the proximal end 34 of the anode body 22. Electrical contact with the anode 20 may be accomplished by in a variety of ways, such as by coupling the lead wire 30 using resistance or laser welding. Alternatively, the lead wire 30 may be embedded into the anode body during its formation (e.g., prior to sintering).

II. Dielectric

A dielectric also overlies or coats the anode body. The dielectric may be formed by anodically oxidizing ("anodizing") the sintered anode so that a dielectric layer is formed over and/or within the anode body. For example, a tantalum (Ta) anode body may be anodized to tantalum pentoxide (Ta₂O₅). Typically, anodization is performed by initially applying a solution to the anode body, such as by dipping the anode body into the electrolyte. A solvent is generally employed, such as water (e.g., deionized water). To enhance ionic conductivity, a compound may be employed that is capable of dissociating in the solvent to form ions. Examples of such compounds include, for instance, acids, such as described below with respect to the electrolyte. For example, an acid (e.g., phosphoric acid) may constitute from about 0.01 wt. % to about 5 wt. %, in some embodiments from about 0.05 wt. % to about 0.8 wt. %, and in some embodiments, from about 0.1 wt. % to about 0.5 wt. % of the anodizing solution. If desired, blends of acids may also be employed.

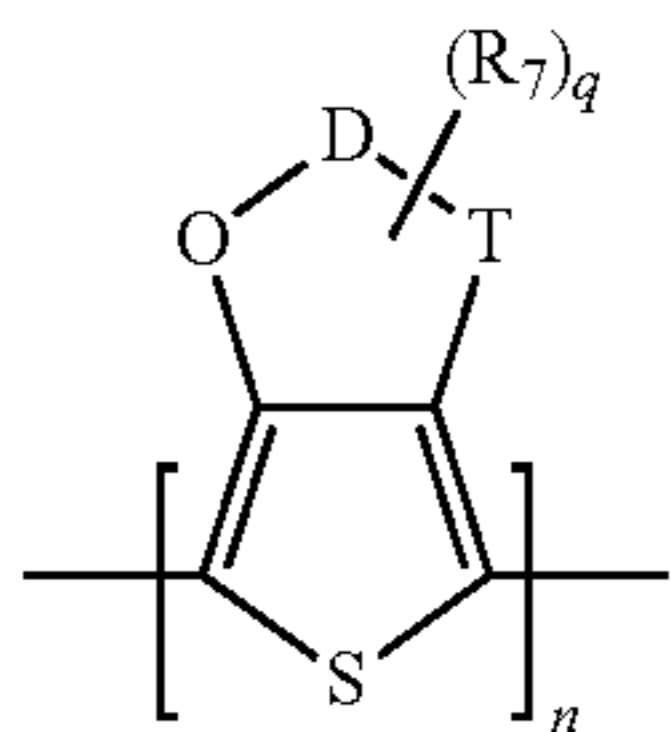
A current is passed through the anodizing solution to form the dielectric layer. The value of the formation voltage manages the thickness of the dielectric layer. For example, the power supply may be initially set up at a galvanostatic mode until the required voltage is reached. Thereafter, the power supply may be switched to a potentiostatic mode to ensure that the desired dielectric thickness is formed over the entire surface of the anode body. Of course, other known methods may also be employed, such as pulse or step potentiostatic

9

methods. The voltage at which anodic oxidation occurs typically ranges from about 4 to about 250 V, and in some embodiments, from about 9 to about 200 V, and in some embodiments, from about 20 to about 150 V. During oxidation, the anodizing solution can be kept at an elevated temperature, such as about 30° C. or more, in some embodiments from about 40° C. to about 200° C., and in some embodiments, from about 50° C. to about 100° C. Anodic oxidation can also be done at ambient temperature or lower. The resulting dielectric layer may be formed on a surface of the anode body and within its pores.

III. Solid Electrolyte

A solid electrolyte overlies the dielectric that generally functions as the cathode for the capacitor. The solid electrolyte contains a conductive polymer, which is typically π -conjugated and has electrical conductivity after oxidation or reduction, such as an electrical conductivity of at least about 1 μ S/cm. Examples of such π -conjugated conductive polymers include, for instance, polyheterocycles (e.g., polypyrroles, polythiophenes, polyanilines, etc.), polyacetylenes, poly-p-phenylenes, polyphenolates, and so forth. In one embodiment, for example, the polymer is a substituted polythiophene, such as those having the following general structure:



wherein,

T is O or S;

D is an optionally substituted C_1 to C_5 alkylene radical (e.g., methylene, ethylene, n-propylene, n-butylene, n-pentylene, etc.);

R_7 is a linear or branched, optionally substituted C_1 to C_{18} alkyl radical (e.g., methyl, ethyl, n- or iso-propyl, n-, iso-, sec- or tert-butyl, n-pentyl, 1-methylbutyl, 2-methylbutyl, 3-methylbutyl, 1-ethylpropyl, 1,1-dimethylpropyl, 1,2-dimethylpropyl, 2,2-dimethylpropyl, n-hexyl, n-heptyl, n-octyl, 2-ethylhexyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-hexadecyl, n-octadecyl, etc.); optionally substituted C_5 to C_{12} cycloalkyl radical (e.g., cyclopentyl, cyclohexyl, cycloheptyl, cyclooctyl, cyclononyl, cyclodecyl, etc.); optionally substituted C_6 to C_{14} aryl radical (e.g., phenyl, naphthyl, etc.); optionally substituted C_7 to C_{18} aralkyl radical (e.g., benzyl, o-, m-, p-tolyl, 2,3-, 2,4-, 2,5-, 2,6-, 3,4-, 3,5-xyllyl, mesityl, etc.); optionally substituted C_1 to C_4 hydroxyalkyl radical, or hydroxyl radical; and

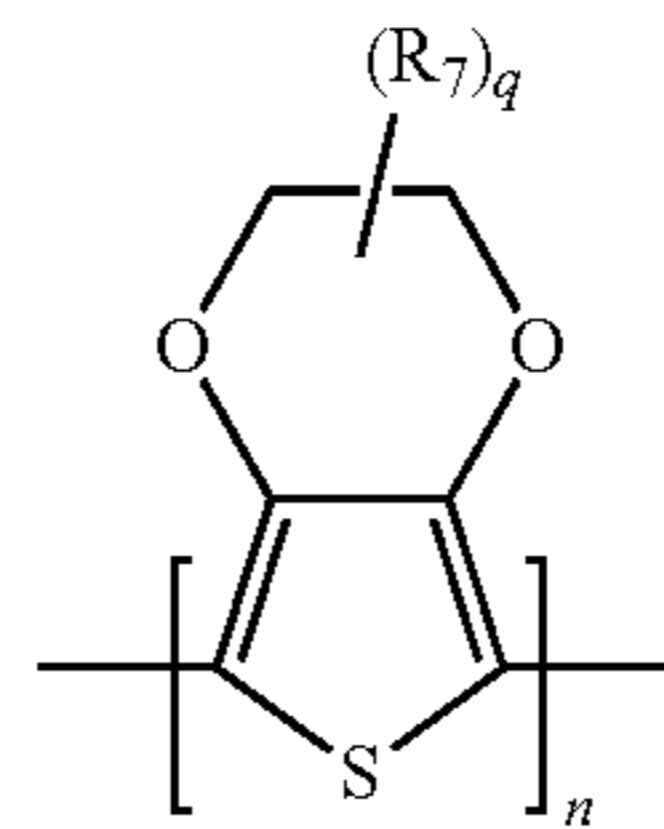
q is an integer from 0 to 8, in some embodiments, from 0 to 2, and in one embodiment, 0; and

n is from 2 to 5,000, in some embodiments from 4 to 2,000, and in some embodiments, from 5 to 1,000. Example of substituents for the radicals "D" or " R_7 " include, for instance, alkyl, cycloalkyl, aryl, aralkyl, alkoxy, halogen, ether, thioether, disulphide, sulfoxide, sulfone, sulfonate, amino, aldehyde, keto, carboxylic acid ester, carboxylic acid, carbonate, carboxylate, cyano, alkylsilane and alkoxy silane groups, carboxylamide groups, and so forth.

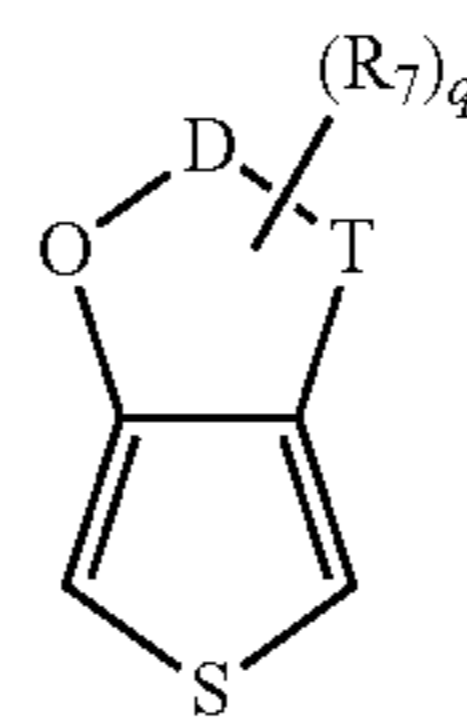
Particularly suitable thiophene polymers are those in which "D" is an optionally substituted C_2 to C_3 alkylene

10

radical. For instance, the polymer may be optionally substituted poly(3,4-ethylenedioxythiophene), which has the following general structure:

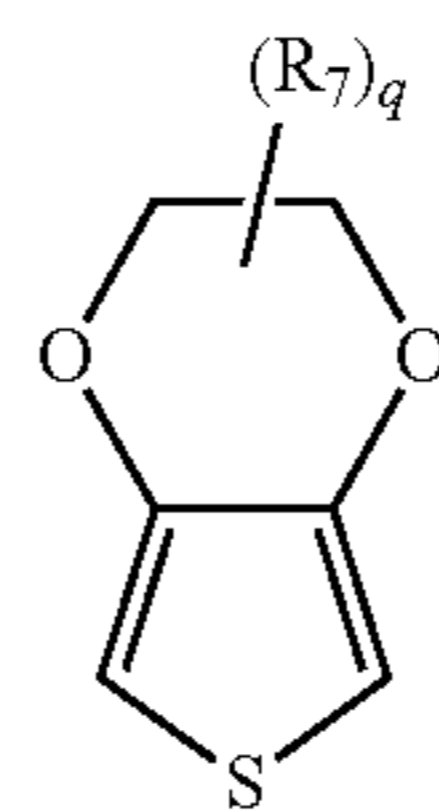


Methods for forming conductive polymers, such as described above, are well known in the art. For instance, U.S. Pat. No. 6,987,663 to Merker, et al., which is incorporated herein in its entirety by reference thereto for all purposes, describes various techniques for forming substituted polythiophenes from a monomeric precursor. The monomeric precursor may, for instance, have the following structure:



wherein,

T, D, R_7 , and q are defined above. Particularly suitable thiophene monomers are those in which "D" is an optionally substituted C_2 to C_3 alkylene radical. For instance, optionally substituted 3,4-alkylenedioxythiophenes may be employed that have the general structure:



wherein, R_7 and q are as defined above. In one particular embodiment, "q" is 0. One commercially suitable example of 3,4-ethylenedioxythiophene is available from H.C. Starck GmbH under the designation Clevios™ M. Other suitable monomers are also described in U.S. Pat. No. 5,111,327 to Blohm, et al. and U.S. Pat. No. 6,635,729 to Groenendaal, et al., which are incorporated herein in their entirety by reference thereto for all purposes. Derivatives of these monomers may also be employed that are, for example, dimers or trimers of the above monomers. Higher molecular derivatives, i.e., tetramers, pentamers, etc. of the monomers are suitable for use in the present invention. The derivatives may be made up of identical or different monomer units and used in pure form and in a mixture with one another and/or with the monomers. Oxidized or reduced forms of these precursors may also be employed.

Regardless of the particular type of polymer, the solid electrolyte includes a plurality of pre-polymerized particles applied in the form of a dispersion. One benefit of employing

a dispersion is that it may minimize the presence of ionic species (e.g., Fe^{2+} or Fe^{3+}) produced during in situ polymerization, which can cause dielectric breakdown under high electric field due to ionic migration. Thus, by applying the conductive polymer as a dispersion rather through in situ polymerization, the resulting capacitor may exhibit a relatively high "breakdown voltage." To enable good impregnation of the anode body, the particles employed in the dispersion typically have a small size, such as an average size (e.g., diameter) of from about 1 to about 150 nanometers, in some embodiments from about 2 to about 50 nanometers, and in some embodiments, from about 5 to about 40 nanometers. The diameter of the particles may be determined using known techniques, such as by ultracentrifuge, laser diffraction, etc. The shape of the particles may likewise vary. In one particular embodiment, for instance, the particles are spherical in shape. However, it should be understood that other shapes are also contemplated by the present invention, such as plates, rods, discs, bars, tubes, irregular shapes, etc. The concentration of the particles in the dispersion may vary depending on the desired viscosity of the dispersion and the particular manner in which the dispersion is to be applied to the capacitor. Typically, however, the particles constitute from about 0.1 to about 10 wt. %, in some embodiments from about 0.4 to about 5 wt. %, and in some embodiments, from about 0.5 to about 4 wt. % of the dispersion.

The dispersion also generally contains a counterion that enhances the stability of the particles. That is, the conductive polymer (e.g., polythiophene or derivative thereof) typically has a charge on the main polymer chain that is neutral or positive (cationic). Polythiophene derivatives, for instance, typically carry a positive charge in the main polymer chain. In some cases, the polymer may possess positive and negative charges in the structural unit, with the positive charge being located on the main chain and the negative charge optionally on the substituents of the radical "R", such as sulfonate or carboxylate groups. The positive charges of the main chain may be partially or wholly saturated with the optionally present anionic groups on the radicals "R." Viewed overall, the polythiophenes may, in these cases, be cationic, neutral or even anionic. Nevertheless, they are all regarded as cationic polythiophenes as the polythiophene main chain has a positive charge.

The counterion may be a monomeric or polymeric anion that counteracts the charge of the conductive polymer. Polymeric anions can, for example, be anions of polymeric carboxylic acids (e.g., polyacrylic acids, polymethacrylic acid, polymaleic acids, etc.); polymeric sulfonic acids (e.g., polystyrene sulfonic acids ("PSS"), polyvinyl sulfonic acids, etc.); and so forth. The acids may also be copolymers, such as copolymers of vinyl carboxylic and vinyl sulfonic acids with other polymerizable monomers, such as acrylic acid esters and styrene. Likewise, suitable monomeric anions include, for example, anions of C_1 to C_{20} alkane sulfonic acids (e.g., dodecane sulfonic acid); aliphatic perfluorosulfonic acids (e.g., trifluoromethane sulfonic acid, perfluorobutane sulfonic acid or perfluorooctane sulfonic acid); aliphatic C_1 to C_{20} carboxylic acids (e.g., 2-ethyl-hexylcarboxylic acid); aliphatic perfluorocarboxylic acids (e.g., trifluoroacetic acid or perfluorooctanoic acid); aromatic sulfonic acids optionally substituted by C_1 to C_{20} alkyl groups (e.g., benzene sulfonic acid, o-toluene sulfonic acid, p-toluene sulfonic acid or dodecylbenzene sulfonic acid); cycloalkane sulfonic acids (e.g., camphor sulfonic acid or tetrafluoroborates, hexafluorophosphates, perchlorates, hexafluoroantimonates, hexafluoroarsenates or hexachloroantimonates); and so forth. Particularly suitable counteranions are polymeric anions, such as a

polymeric carboxylic or sulfonic acid (e.g., polystyrene sulfonic acid ("PSS")). The molecular weight of such polymeric anions typically ranges from about 1,000 to about 2,000,000, and in some embodiments, from about 2,000 to about 500,000.

When employed, the weight ratio of such counterions to conductive polymers in the dispersion and in the resulting layer is typically from about 0.5:1 to about 50:1, in some embodiments from about 1:1 to about 30:1, and in some embodiments, from about 2:1 to about 20:1. The weight of the electrically conductive polymers corresponds referred to the above-referenced weight ratios refers to the weighed-in portion of the monomers used, assuming that a complete conversion occurs during polymerization.

In addition to conductive polymer(s) and counterion(s), the dispersion may also contain one or more binders to further enhance the adhesive nature of the polymeric layer and also increase the stability of the particles within the dispersion. The binders may be organic in nature, such as polyvinyl alcohols, polyvinyl pyrrolidones, polyvinyl chlorides, polyvinyl acetates, polyvinyl butyrates, polyacrylic acid esters, polyacrylic acid amides, polymethacrylic acid esters, polymethacrylic acid amides, polyacrylonitriles, styrene/acrylic acid ester, vinyl acetate/acrylic acid ester and ethylene/vinyl acetate copolymers, polybutadienes, polyisoprenes, polystyrenes, polyethers, polyesters, polycarbonates, polyurethanes, polyamides, polyimides, polysulfones, melamine formaldehyde resins, epoxide resins, silicone resins or celluloses. Crosslinking agents may also be employed to enhance the adhesion capacity of the binders. Such crosslinking agents may include, for instance, melamine compounds, masked isocyanates or functional silanes, such as 3-glycidoxypropyltrialkoxysilane, tetraethoxysilane and tetraethoxysilane hydrolysate or crosslinkable polymers, such as polyurethanes, polyacrylates or polyolefins, and subsequent crosslinking.

Dispersion agents may also be employed to facilitate the formation of the solid electrolyte and the ability to apply it to the anode part. Suitable dispersion agents include solvents, such as aliphatic alcohols (e.g., methanol, ethanol, propanol and butanol), aliphatic ketones (e.g., acetone and methyl ethyl ketones), aliphatic carboxylic acid esters (e.g., ethyl acetate and butyl acetate), aromatic hydrocarbons (e.g., toluene and xylene), aliphatic hydrocarbons (e.g., hexane, heptane and cyclohexane), chlorinated hydrocarbons (e.g., dichloromethane and dichloroethane), aliphatic nitriles (e.g., acetonitrile), aliphatic sulfoxides and sulfones (e.g., dimethyl sulfoxide and sulfolane), aliphatic carboxylic acid amides (e.g., methylacetamide, dimethylacetamide and dimethylformamide), aliphatic and araliphatic ethers (e.g., diethylether and anisole), water, and mixtures of any of the foregoing solvents. A particularly suitable dispersion agent is water.

In addition to those mentioned above, still other ingredients may also be used in the dispersion. For example, conventional fillers may be used that have a size of from about 10 nanometers to about 100 micrometers, in some embodiments from about 50 nanometers to about 50 micrometers, and in some embodiments, from about 100 nanometers to about 30 micrometers. Examples of such fillers include calcium carbonate, silicates, silica, calcium or barium sulfate, aluminum hydroxide, glass fibers or bulbs, wood flour, cellulose powder carbon black, electrically conductive polymers, etc. The fillers may be introduced into the dispersion in powder form, but may also be present in another form, such as fibers.

Surface-active substances may also be employed in the dispersion, such as ionic or non-ionic surfactants. Furthermore, adhesives may be employed, such as organofunctional

silanes or their hydrolysates, for example 3-glycidoxypropyltrialkoxysilane, 3-aminopropyl-triethoxysilane, 3-mercaptopropyl-trimethoxysilane, 3-metacryloxypropyltrimethoxysilane, vinyltrimethoxysilane or octyltriethoxysilane. The dispersion may also contain additives that increase conductivity, such as ether group-containing compounds (e.g., tetrahydrofuran), lactone group-containing compounds (e.g., γ -butyrolactone or γ -valerolactone), amide or lactam group-containing compounds (e.g., caprolactam, N-methylcaprolactam, N,N-dimethylacetamide, N-methylacetamide, N,N-dimethylformamide (DMF), N-methylformamide, N-methylformanilide, N-methylpyrrolidone (NMP), N-octylpyrrolidone, or pyrrolidone), sulfones and sulfoxides (e.g., sulfolane (tetramethylenesulfone) or dimethylsulfoxide (DMSO)), sugar or sugar derivatives (e.g., saccharose, glucose, fructose, or lactose), sugar alcohols (e.g., sorbitol or mannitol), furan derivatives (e.g., 2-furancarboxylic acid or 3-furancarboxylic acid), an alcohols (e.g., ethylene glycol, glycerol, di- or triethylene glycol).

The polymeric dispersion may be applied using a variety of known techniques, such as by spin coating, impregnation, pouring, dropwise application, injection, spraying, doctor blading, brushing, printing (e.g., ink-jet, screen, or pad printing), or dipping. Although it may vary depending on the application technique employed, the viscosity of the dispersion is typically from about 0.1 to about 100,000 mPas (measured at a shear rate of 100 s^{-1}), in some embodiments from about 1 to about 10,000 mPas, in some embodiments from about 10 to about 1,500 mPas, and in some embodiments, from about 100 to about 1000 mPas. Once applied, the layer may be dried and/or washed. One or more additional layers may also be formed in this manner to achieve the desired thickness. Typically, the total thickness of the layer(s) formed by this particle dispersion is from about 1 to about $50 \mu\text{m}$, and in some embodiments, from about 5 to about $20 \mu\text{m}$. The weight ratio of counterions to conductive polymers is likewise from about 0.5:1 to about 50:1, in some embodiments from about 1:1 to about 30:1, and in some embodiments, from about 2:1 to about 20:1.

In addition to pre-polymerized particles, the solid electrolyte may optionally contain other components, such as a hydroxy-functional nonionic polymer. The term "hydroxy-functional" generally means that the compound contains at least one hydroxyl functional group or is capable of possessing such a functional group in the presence of a solvent. Without intending to be limited by theory, it is believed that hydroxy-functional nonionic polymers can improve the degree of contact between the polymer particles and the surface of the internal dielectric, which is typically relatively smooth in nature as a result of higher forming voltages. This unexpectedly increases the breakdown voltage and wet-to-dry capacitance of the resulting capacitor. Furthermore, it is believed that the use of a hydroxy-functional polymer with a certain molecular weight can also minimize the likelihood of chemical decomposition at high voltages. For instance, the molecular weight of the hydroxy-functional polymer may be from about 100 to 10,000 grams per mole, in some embodiments from about 200 to 2,000, in some embodiments from about 300 to about 1,200, and in some embodiments, from about 400 to about 800.

Any of a variety of hydroxy-functional nonionic polymers may generally be employed for this purpose. In one embodiment, for example, the hydroxy-functional polymer is a polyalkylene ether. Polyalkylene ethers may include polyalkylene glycols (e.g., polyethylene glycols, polypropylene glycols, polytetramethylene glycols, polyepichlorohydrins, etc.), polyoxetanes, polyphenylene ethers, polyether ketones, and

so forth. Polyalkylene ethers are typically predominantly linear, nonionic polymers with terminal hydroxy groups. Particularly suitable are polyethylene glycols, polypropylene glycols and polytetramethylene glycols (polytetrahydrofurans), which are produced by polyaddition of ethylene oxide, propylene oxide or tetrahydrofuran onto water. The polyalkylene ethers may be prepared by polycondensation reactions from diols or polyols. The diol component may be selected, in particular, from saturated or unsaturated, branched or unbranched, aliphatic dihydroxy compounds containing 5 to 36 carbon atoms or aromatic dihydroxy compounds, such as, for example, pentane-1,5-diol, hexane-1,6-diol, neopentyl glycol, bis-(hydroxymethyl)-cyclohexanes, bisphenol A, dimer diols, hydrogenated dimer diols or even mixtures of the diols mentioned. In addition, polyhydric alcohols may also be used in the polymerization reaction, including for example glycerol, di- and polyglycerol, trimethylolpropane, pentaerythritol or sorbitol.

In addition to those noted above, other hydroxy-functional nonionic polymers may also be employed in the present invention. Some examples of such polymers include, for instance, ethoxylated alkylphenols; ethoxylated or propoxylated C_6 - C_{24} fatty alcohols; polyoxyethylene glycol alkyl ethers having the general formula: $\text{CH}_3-(\text{CH}_2)_{10-16}-(\text{O}-\text{C}_2\text{H}_4)_{1-25}-\text{OH}$ (e.g., octaethylene glycol monododecyl ether and pentaethylene glycol monododecyl ether); polyoxypropylene glycol alkyl ethers having the general formula: $\text{CH}_3-(\text{CH}_2)_{10-16}-(\text{O}-\text{C}_3\text{H}_6)_{1-25}-\text{OH}$; polyoxyethylene glycol octylphenol ethers having the following general formula: $\text{C}_8\text{H}_{17}-(\text{C}_6\text{H}_4)-(\text{O}-\text{C}_2\text{H}_4)_{1-25}-\text{OH}$ (e.g., Triton™ X-100); polyoxyethylene glycol alkylphenol ethers having the following general formula: $\text{C}_9\text{H}_{19}-(\text{C}_6\text{H}_4)-(\text{O}-\text{C}_2\text{H}_4)_{1-25}-\text{OH}$ (e.g., nonoxynol-9); polyoxyethylene glycol esters of C_8 - C_{24} fatty acids, such as polyoxyethylene glycol sorbitan alkyl esters (e.g., polyoxyethylene (20) sorbitan monolaurate, polyoxyethylene (20) sorbitan monopalmitate, polyoxyethylene (20) sorbitan monostearate, polyoxyethylene (20) sorbitan monooleate, PEG-20 methyl glucose distearate, PEG-20 methyl glucose sesquisteate, PEG-80 castor oil, and PEG-20 castor oil, PEG-3 castor oil, PEG 600 dioleate, and PEG 400 dioleate) and polyoxyethylene glycerol alkyl esters (e.g., polyoxyethylene-23 glycerol laurate and polyoxyethylene-20 glycerol stearate); polyoxyethylene glycol ethers of C_8 - C_{24} fatty acids (e.g., polyoxyethylene-10 cetyl ether, polyoxyethylene-10 stearyl ether, polyoxyethylene-20 cetyl ether, polyoxyethylene-10 oleyl ether, polyoxyethylene-20 oleyl ether, polyoxyethylene-20 isohexadecyl ether, polyoxyethylene-15 tridecyl ether, and polyoxyethylene-6 tridecyl ether); block copolymers of polyethylene glycol and polypropylene glycol (e.g., Poloxamers); and so forth, as well as mixtures thereof.

The hydroxy-functional nonionic polymer may be incorporated into the solid electrolyte in a variety of different ways. In certain embodiments, for instance, the hydroxy-functional polymer may simply be incorporated into any layer(s) formed by the initial dispersion described above. In such embodiments, the concentration of the hydroxy-functional polymer in the dispersion is typically from about 1 wt. % to about 50 wt. %, in some embodiments from about 5 wt. % to about 40 wt. %, and in some embodiments, from about 10 wt. % to about 30 wt. %.

In other embodiments, however, the hydroxy-functional polymer may be applied after the initial polymer dispersion is applied to the anode body. In such embodiments, the technique used to apply the hydroxy-functional polymer may vary. For example, the polymer may be applied in the form of a liquid solution using various methods, such as immersion,

dipping, pouring, dripping, injection, spraying, spreading, painting or printing, for example, inkjet, screen printing or tampon printing. Solvents known to the person skilled in the art can be employed in the solution, such as water, alcohols, or a mixture thereof. The concentration of the hydroxy-functional polymer in such a solution typically ranges from about 5 wt % to about 95 wt. %, in some embodiments from about 10 wt. % to about 70 wt. %, and in some embodiments, from about 15 wt. % to about 50 wt. % of the solution. If desired, such solutions may be generally free of conductive polymers. For example, conductive polymers may constitute about 2 wt % or less, in some embodiments about 1 wt. % or less, and in some embodiments, about 0.5 wt. % or less of the solution.

Alternatively, however, it may also be desired to employ a conductive polymer in combination with the hydroxy-functional polymer. For example, in certain embodiments, a second polymer dispersion that contains conductive polymer particles and a hydroxy-functional polymer is applied to the anode body after the initial layer(s) formed from the first polymer dispersion are applied to the anode body. The conductive particles of the second polymer dispersion are generally described above, although they need not be identical to those employed in the first polymer dispersion. For instance, the first and second polymer dispersion may employ particles of a different size and/or chemical composition. Regardless, the concentration of the hydroxy-functional polymer in the second polymer dispersion is typically from about 1 wt. % to about 50 wt. %, in some embodiments from about 5 wt. % to about 40 wt. %, and in some embodiments, from about 10 wt. % to about 30 wt. %. Likewise, in those embodiments in which the hydroxy-functional polymer is employed in a second dispersion, it may also be desirable that the first dispersion is generally free of such hydroxy-functional nonionic polymers. For example, hydroxy-functional polymers may constitute about 2 wt. % or less, in some embodiments about 1 wt. % or less, and in some embodiments, about 0.5 wt. % or less of the first polymer dispersion. Once applied, the layer formed by the second polymer dispersion may be dried and/or washed. One or more additional layers may also be formed in this manner to achieve the desired thickness. Typically, the total thickness of the layers formed by the second polymer dispersion is from about 0.1 to about 5 μm , in some embodiments from about 0.1 to about 3 μm , and in some embodiments, from about 0.2 to about 1 μm .

IV. External Polymer Coating

Although not required, an external polymer coating may also be applied to the anode body and overlie the solid electrolyte. The external polymer coating generally contains one or more layers formed from a dispersion of pre-polymerized conductive particles, such as described in more detail above. The external coating may be able to further penetrate into the edge region of the capacitor body to increase the adhesion to the dielectric and result in a more mechanically robust part, which may reduce equivalent series resistance and leakage current. Because it is generally intended to improve the degree of edge coverage rather than impregnate the interior of the anode body, the particles used in the external coating typically have a larger size than those employed in any optional dispersions of the solid electrolyte. For example, the ratio of the average size of the particles employed in the external polymer coating to the average size of the particles employed in any dispersion of the solid electrolyte is typically from about 1.5 to about 30, in some embodiments from about 2 to about 20, and in some embodiments, from about 5 to about 15. For example, the particles employed in the dispersion of the external coating may have an average size of from about 50 to about 500 nanometers, in some embodiments

from about 80 to about 250 nanometers, and in some embodiments, from about 100 to about 200 nanometers.

If desired, a crosslinking agent may also be employed in the external polymer coating to enhance the degree of adhesion to the solid electrolyte. Typically, the crosslinking agent is applied prior to application of the dispersion used in the external coating. Suitable crosslinking agents are described, for instance, in U.S. Patent Publication No. 2007/0064376 to Merker, et al. and include, for instance, amines (e.g., diamines, triamines, oligomer amines, polyamines, etc.); polyvalent metal cations, such as salts or compounds of Mg, Al, Ca, Fe, Cr, Mn, Ba, Ti, Co, Ni, Cu, Ru, Ce or Zn, phosphonium compounds, sulfonium compounds, etc. Particularly suitable examples include, for instance, 1,4-diaminocyclohexane, 1,4-bis(amino-methyl)cyclohexane, ethylenediamine, 1,6-hexanediamine, 1,7-heptanediamine, 1,8-octanediamine, 1,9-nonanediamine, 1,10-decanediamine, 1,12-dodecanediamine, N,N-dimethylethylenediamine, N,N,N',N'-tetramethylethylenediamine, N,N,N',N'-tetramethyl-1,4-butanediamine, etc., as well as mixtures thereof.

The crosslinking agent is typically applied from a solution or dispersion whose pH is from 1 to 10, in some embodiments from 2 to 7, in some embodiments, from 3 to 6, as determined at 25°C. Acidic compounds may be employed to help achieve the desired pH level. Examples of solvents or dispersants for the crosslinking agent include water or organic solvents, such as alcohols, ketones, carboxylic esters, etc. The crosslinking agent may be applied to the capacitor body by any known process, such as spin-coating, impregnation, casting, dropwise application, spray application, vapor deposition, sputtering, sublimation, knife-coating, painting or printing, for example inkjet, screen or pad printing. Once applied, the crosslinking agent may be dried prior to application of the polymer dispersion. This process may then be repeated until the desired thickness is achieved. For example, the total thickness of the entire external polymer coating, including the crosslinking agent and dispersion layers, may range from about 1 to about 50 μm , in some embodiments from about 2 to about 40 μm , and in some embodiments, from about 5 to about 20 μm .

V. Other Components of the Capacitor

If desired, the capacitor may also contain other layers as is known in the art. For example, a protective coating may optionally be formed between the dielectric and solid electrolyte, such as one made of a relatively insulative resinous material (natural or synthetic). Such materials may have a specific resistivity of greater than about 10 $\Omega\cdot\text{cm}$, in some embodiments greater than about 100, in some embodiments greater than about 1,000 $\Omega\cdot\text{cm}$, in some embodiments greater than about 1×10^5 $\Omega\cdot\text{cm}$, and in some embodiments, greater than about 1×10^{10} $\Omega\cdot\text{cm}$. Some resinous materials that may be utilized in the present invention include, but are not limited to, polyurethane, polystyrene, esters of unsaturated or saturated fatty acids (e.g., glycerides), and so forth. For instance, suitable esters of fatty acids include, but are not limited to, esters of lauric acid, myristic acid, palmitic acid, stearic acid, eleostearic acid, oleic acid, linoleic acid, linolenic acid, aleuritic acid, shellolic acid, and so forth. These esters of fatty acids have been found particularly useful when used in relatively complex combinations to form a "drying oil", which allows the resulting film to rapidly polymerize into a stable layer. Such drying oils may include mono-, di-, and/or triglycerides, which have a glycerol backbone with one, two, and three, respectively, fatty acyl residues that are esterified. For instance, some suitable drying oils that may be used include, but are not limited to, olive oil, linseed oil, castor oil,

tung oil, soybean oil, and shellac. These and other protective coating materials are described in more detail U.S. Pat. No. 6,674,635 to Fife, et al., which is incorporated herein in its entirety by reference thereto for all purposes.

If desired, the part may also be applied with a carbon layer (e.g., graphite) and silver layer, respectively. The silver coating may, for instance, act as a solderable conductor, contact layer, and/or charge collector for the capacitor and the carbon coating may limit contact of the silver coating with the solid electrolyte. Such coatings may cover some or all of the solid electrolyte.

The particular manner in which the components are incorporated into the capacitor is not critical and may be accomplished using a variety of techniques. For example, a capacitor assembly may be formed that contains a casing within which the capacitor is contained. The casing may have any of a variety of shapes, such as generally cylindrical, rectangular, triangular, prismatic, etc. The casing may be formed from a variety of materials, such as a ceramic, metal, e.g., tantalum, niobium, aluminum, nickel, hafnium, titanium, copper, silver, steel (e.g., stainless), alloys thereof (e.g., electrically conductive oxides), composites thereof (e.g., metal coated with electrically conductive oxide), etc., as well as other materials. If desired, the capacitor may also be hermetically sealed within the casing in the presence of a gaseous atmosphere that contains an inert gas, such as argon, nitrogen, etc. The casing may optionally include a lid that covers, which may be formed from the same or different material than the casing. The lid or casing may define an internal orifice through which extends a conductive tube that is generally hollow and of a size and shape sufficient to accommodate the anode lead. The conductive tube is typically formed from a metal, such as tantalum, niobium, aluminum, nickel, hafnium, titanium, copper, silver, steel (e.g., stainless), alloys thereof (e.g., electrically conductive oxides), composites thereof (e.g., metal coated with electrically conductive oxide), and so forth.

The resulting capacitor may maintain a low equivalence series resistance ("ESR"), such as less than about 100 mohms, in some embodiments less than about 75 mohms, in some embodiments from about 0.01 to about 60 mohms, and in some embodiments, from about 0.05 to about 50 mohms, measured at an operating frequency of 100 kHz. In certain cases, such improved capacitance and ESR performance can remain stable under a variety of different conditions. For example, the capacitance and/or equivalent series resistance of the capacitor may be within the ranges noted above even at low temperatures, such as about 25° C. or less, in some embodiments about 10° C. or less, in some embodiments about 0° C. or less, and in some embodiments, from about -75° C. to about -25° C. (e.g., -55° C.), as well as at a wide range of frequencies, such from about 10 Hz to about 100 kHz. The leakage current, which generally refers to the current flowing from one conductor to an adjacent conductor through an insulator, can also be maintained at relatively low levels. For example, the numerical value of the normalized leakage current of a capacitor of the present invention is, in some embodiments, less than about 1 $\mu\text{A}/\mu\text{F}\cdot\text{V}$, in some embodiments less than about 0.5 $\mu\text{A}/\mu\text{F}\cdot\text{V}$, and in some embodiments, less than about 0.1 $\mu\text{A}/\mu\text{F}\cdot\text{V}$, where μA is microamps and $\mu\text{F}\cdot\text{V}$ is the product of the capacitance and the rated voltage. Such normalized leakage current values may even be maintained after aging for a substantial amount of time at high temperatures. For example, the values may be maintained for about 100 hours or more, in some embodiments from about 300 hours to about 3000 hours, and in some embodiments, from about 400 hours to about 2500 hours (e.g., 500 hours, 600 hours, 700 hours, 800 hours, 900 hours,

1000 hours, 1100 hours, 1200 hours, or 2000 hours) at temperatures ranging from about -55° C. to about 250° C., in some embodiments from about 0° C. to about 225° C., and in some embodiments, from about 10° C. to about 225° C.

The present invention may be better understood by reference to the following examples.

Test Procedures

Capacitance ("Cap")

The capacitance was measured via 'wet measurement' using a Keithley 3330 Precision LCZ meter with Kelvin Leads with 0 volt DC bias and a 1.0 volt peak to peak sinusoidal signal. The operating frequency was 120 Hz and the temperature was 23° C. \pm 2° C. The capacitance was measured in 5M aqueous sulfuric acid electrolyte when one anode was set-up as anode and second anode was set-up as cathode. The measured value was multiplied by two to achieve the average capacitance of both anodes.

EXAMPLE 1

70,000 $\mu\text{FV/g}$ tantalum powder was used to form anode samples. Each anode sample was embedded with a tantalum wire, sintered at 1450° C., and pressed into a cylindrically-shaped anode having a density of 5.6 g/cm³ and containing five (5) symmetrical longitudinally extending channels recessed into the anode body. The resulting cylindrical part had a size of 20.44 (height) \times 8.48 (diameter) mm and weight of 6.143 g. The part was anodized to 75V in water/phosphoric acid electrolyte with conductivity 8.6 mS at temperature of 85° C. to form the dielectric layer. A conductive polymer coating was then formed by vacuum dipping the anode into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 1.1% and viscosity 20 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the parts were dried at 125° C. for 20 minutes. This process was repeated 10 times. Thereafter, the part was vacuum dipped into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 2% and viscosity 20 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the part was dried at 125° C. for 20 minutes. This process was repeated 3 times. Thereafter, the part was dipped into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 2% and viscosity 160 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the parts were dried at 125° C. for 20 minutes. This process was repeated 8 times. The parts were then dipped into a graphite dispersion and dried. Finally, the part was dipped into a silver dispersion and dried. Multiple parts (10) of 3000 $\mu\text{F}/50\text{V}$ capacitors were made in this manner.

EXAMPLE 2

A capacitor was formed in the manner described in Example 1, except using a different conductive polymer coating. The conductive polymer coating was formed by vacuum dipping the anode into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 1.1% and viscosity 20 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the part was dried at 125° C. for 20 minutes. This process was repeated 10 times. Thereafter, the part was vacuum dipped into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 2% and viscosity 20 mPa·s (Clevios™ K, H.C. Starck) and additional 20% solids content of poly(ethylene glycol) with molecular weight 600 (Sigma Aldrich®). Upon coating, the part was dried at 125° C. for 20 minutes. This process was not repeated. Thereafter, the part was dipped into a dispersed poly(3,4-ethylenedioxythiophene) having a sol-

19

ids content 2% and viscosity 160 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the part was dried at 125° C. for 20 minutes. This process was repeated 8 times. The part was then dipped into a graphite dispersion and dried. Finally, the part was dipped into a silver dispersion and dried. Multiple parts (10) of 3000 μF/50V capacitors were made in this manner.

EXAMPLE 3

A capacitor was formed in the manner described in Example 1, except using a different conductive polymer coating. The conductive polymer coating was formed by vacuum dipping the anode into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 1.1% and viscosity 20 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the parts were dried at 125° C. for 20 minutes. This process was repeated 10 times. Thereafter, the part was vacuum dipped into a aqueous solution having a solids content 20% of poly(ethylene glycol) with a molecular weight of 600 (Sigma Aldrich). Upon coating, the part was dried at 125° C. for 20 minutes. This process was not repeated. Thereafter, the part was dipped into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 2% and viscosity 20 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the part was dried at 125° C. for 20 minutes. This process was not repeated. Thereafter, the parts were dipped into a dispersed poly(3,4-ethylenedioxythiophene) having a solids content 2% and viscosity 160 mPa·s (Clevios™ K, H.C. Starck). Upon coating, the part was dried at 125° C. for 20 minutes. This process was repeated 8 times. The part was then dipped into a graphite dispersion and dried. Finally, the part was dipped into a silver dispersion and dried. Multiple parts (10) of 3000 μF/50V capacitors were made in this manner.

COMPARATIVE EXAMPLE

Capacitors were formed in the manner described in Example 1, except that the pressed anode had a weight of 7.511 g and lacked any channels. Multiple parts (10) of 3000 μF/50V capacitors were made in this manner.

The finished capacitors of Examples 1-3 and the Comparative Example were then tested for electrical performance before assembly process. The median results of capacitance, and ESR are set forth also below in Table 1. The wet capacitance was 2955 μF for Examples 1-3 and 4140 μF for the Comparative Example.

TABLE 1

	Electrical Properties		
	Dry Cap [μF]	Wet-to-Dry Cap [%]	ESR [mΩ]
Example 1	2113.3	71.52	25.8
Example 2	2203.0	74.55	27.2
Example 3	2222.3	75.20	23.1
Comparative Example	2851.6	68.86	29.4

As indicated, the parts formed as described herein had a relatively low ESR and high dry/wet capacitance.

These and other modifications and variations of the present invention may be practiced by those of ordinary skill in the art, without departing from the spirit and scope of the present invention. In addition, it should be understood that aspects of the various embodiments may be interchanged both in whole or in part. Furthermore, those of ordinary skill in the art will appreciate that the foregoing description is by way of

20

example only, and is not intended to limit the invention so further described in such appended claims.

What is claimed is:

1. A solid electrolytic capacitor comprising:

an anode that comprises a sintered porous anode body extending in a longitudinal direction, wherein the anode body contains a sidewall positioned between a proximal end and an opposing distal end, wherein a longitudinally extending channel is recessed into the sidewall; a dielectric overlying the anode body; and a solid electrolyte overlying the dielectric, wherein the solid electrolyte comprises a plurality of pre-polymerized conductive polymer particles.

2. The solid electrolytic capacitor of claim 1, wherein the anode body is formed from a powder that contains tantalum, niobium, or an electrically conductive oxide thereof.

3. The solid electrolytic capacitor of claim 1, wherein the anode body has a length in the longitudinal direction and a width and depth, and wherein the ratio of the length to both the width and depth is from about 1.1 to about 10.

4. The solid electrolytic capacitor of claim 1, wherein the longitudinally extending channel intersects both the distal end and the proximal end.

5. The solid electrolytic capacitor of claim 1, wherein an anode lead extends from the proximal end of the anode in the longitudinal direction.

6. The solid electrolytic capacitor of claim 1, wherein the anode body has a generally cylindrical shape.

7. The solid electrolytic capacitor of claim 1, wherein the channel has an aspect ratio of from about 15 to about 150.

8. The solid electrolytic capacitor of claim 1, wherein the channel has a length of from about 1 to about 60 millimeters and a width of from about 0.02 to about 15 millimeters.

9. The solid electrolytic capacitor of claim 1, wherein the channel has a substantially constant width.

10. The solid electrolytic capacitor of claim 1, wherein the channel has a depth that is from about 5% to about 50% of the thickness of the porous anode body.

11. The solid electrolytic capacitor of claim 1, wherein a plurality of longitudinally extending channels are recessed into the sidewall of the anode body.

12. The solid electrolytic capacitor of claim 11, wherein the channels are distributed in a generally symmetrical manner about a longitudinal center of the anode body.

13. The solid electrolytic capacitor of claim 11, wherein the anode body contains first and second longitudinally extending channels, the first channels being recessed into the sidewall to a different depth than the second channels.

14. The solid electrolytic capacitor of claim 1, wherein the channel is substantially parallel to the longitudinal direction.

15. The solid electrolytic capacitor of claim 1, wherein the pre-polymerized particles are formed from poly(3,4-ethylenedioxythiophene).

16. The solid electrolytic capacitor of claim 1, wherein the pre-polymerized particles have an average size of from about 2 to about 50 nanometers.

17. The solid electrolytic capacitor of claim 1, wherein the solid electrolyte further comprises a hydroxy-functional non-ionic copolymer.

18. The solid electrolytic capacitor of claim 1, wherein the capacitor exhibits a breakdown voltage of about 60 volts or more and a wet-to-dry capacitance of about 50% or more.

19. A capacitor assembly comprising a casing within which the capacitor of claim 1 is hermetically sealed.

20. A method for forming the solid electrolytic capacitor of claim 1, the method comprising:

21

pressing and sintering a powder to form a sintered porous body containing a longitudinally extending channel recessed into a sidewall positioned between a proximal end and an opposing distal end;
anodically oxidizing the sintered porous body to form a dielectric that overlies the anode; and
applying a dispersion of pre-polymerized conductive polymer particles to the dielectric.

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22